

PATENT APPLICATION Mo-7309-US 06160-1P67

## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

APPLICATION OF		) ) GROUP NO.: 1742 )
PETER R. JEPSON ET AL		
SERIAL NUMBER: 10/079,286		) EXAMINER: LOIS L. ZHENG
FILED:	FEBRUARY 20, 2002	
TITLE:	REFRACTORY METAL PLATES WITH UNIFORM TEXTURE	

### **DECLARATION UNDER 37 C.F.R. § 1.132**

- I, Peter R. Jepson, residing at 21 Marsh Avenue, Newbury, MA, 01951, United States, declare as follows.
- 1) That I have the following technical education and experience:
  - a) I am a metallurgist having studied at Cambridge University, England, from 1969 to 1972.
  - b) I received the degrees of: bachelor of arts at Cambridge University in the year of 1972; and master of arts at Cambridge University in the year of 1976.
  - c) I am presently employed by H.C. Starck Inc., 45 Industrial Place,
    Newton, Massachusetts, 02461-1951, United States, and have been
    so employed since October 1998, in the research department, in
    particular with regard to the development of novel metal-working
    processes.
- 2) That the following tests were carried out under my immediate supervision and control.

## **Process of Manufacture**

Per the description at paragraph [0040] in United States Patent Application Publication No. US 2002/0072475 A1 (*Michaluk '475*), an EB-melted tantalum (rather than niobium) ingot was processed into a sputtering-target plate. The tantalum plate prepared in accordance with the disclosure of *Michaluk '47*5 is referred to herein as Plate 475. As used herein, the term "EB" means "electron beam."

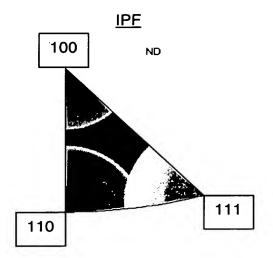
In accordance with the description at paragraphs 0026, 0028 and 0029 of Applicants' present specification, an EB-melted tantalum plate according to Applicants' claimed invention was processed into a sputtering-target plate. The tantalum plate according to Applicants' present claims is referred herein as Plate 286.

### **Texture Measurements**

Three samples were cut along the length of each of the plates, and the texture examined on a cross-sectional (through-thickness) face, using an EBSD method, as described in 'Microtexture Determination' by V. Randle, Maney Publishing, 2003. For each sample, a grain map was generated. The color scheme of the grain maps is shown in Inverse Pole Figure ("IPF") format, in which the colors represent the orientation of each analyzed point, and each grain, relative to the plate Normal Direction ("ND"). Note, however, that what is black on the IPF is grey on the maps. The map extends from one surface of the plate (top of the map) to the other surface of the plate (bottom of the map). The thickness of Plate 475 was about 7 mm, and that of Plate 286 about 6 mm. The length of each plate sample examined, in each case (i.e., for Plate 475 and Plate 286), was about 1.5 mm.

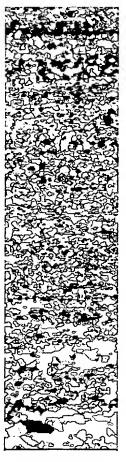
Plate 475 and Plate 286 were in each case observed to possess some thickness variation across each plate, and thickness tapering towards the edge of each plate. In the following presentation of texture results, Samples A through C of Plate 475 show less thickness tapering than those of Plate 268, due to the variation in thickness across the plates (i.e., the three samples cut from Plate 475 happened to have a very similar thickness due to the thickness variation across the plate).

## **Texture Results**

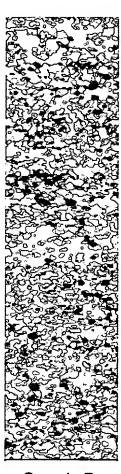


## Texture Results (cont'd):

# <u>Plate 475</u>





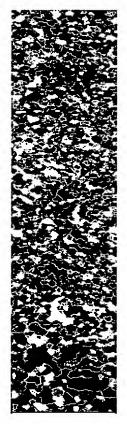


Sample B



## Texture Results (cont'd):

# <u>Plate 286</u>



Sample A



Sample B



Sample C

#### Comments

The samples from Plate 475, when analyzed in accordance with the method described in paragraph [0030] of *Michaluk '475*, were found to have an Ln ratio of {111}:{100} center peak intensities within the same increment of greater than about -4.0, and {100} peak center intensities less than 15 random. That is, what *Michaluk '475* describes as "homogenous texture" clearly is not homogenous, in particular when compared to the uniform texture of tantalum plate according to Applicants' present claims.

It can be clearly seen that, in the case of Plate 475, the texture varies substantially from sample to sample at any given depth from the top surface (i.e., across the surface of any plane of the plate), whereas in the case of Plate 286, the texture of the three samples is very similar (i.e., across the surface of any plane of the plate). In addition, the samples from Plate 475 have non-uniform texture through the thicknesses thereof, while the samples from Plate 286 have texture that is noticeably more uniform through the thickness thereof. In other words, with Plate 475, the distribution of {100} and {111} crystallographic orientations varies greatly (i.e., is non-uniform / non-homogenous) across the surface of any plane of the plate, and through any thickness of the plate. Whereas with Plate 286, the distribution of {100} and {111} crystallographic orientations varies only slightly (i.e., is substantially uniform / homogenous) across the surface of any plane of the plate, and through any thickness of the plate.

I further declare that all statements made herein are of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States code and that such willful false statements may jeopardize the validity of pending Application Serial Number 10/079,286 or any patent issuing thereon.

Signed at Newton, MA this 16<sup>th</sup> day of November, 2005.

Peter R. Jepson

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